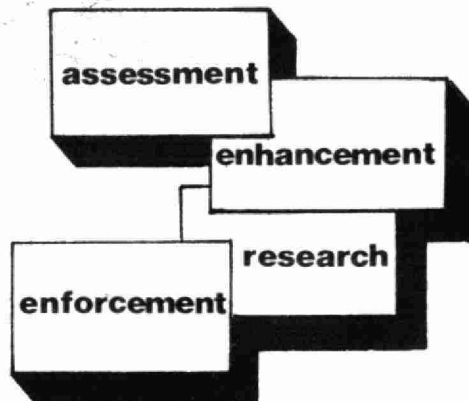
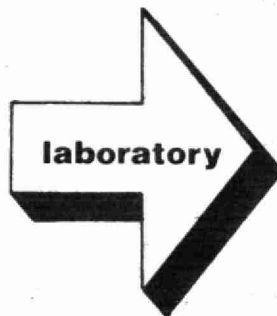
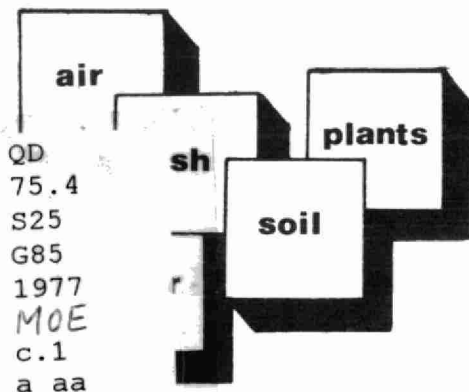


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A GUIDE TO THE COLLECTION AND SUBMISSION OF SAMPLES FOR LABORATORY ANALYSIS



MINISTRY
OF THE
ENVIRONMENT

G.C. Ronan
Director
Laboratory Services Branch

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A GUIDE TO THE COLLECTION AND SUBMISSION
OF
SAMPLES FOR LABORATORY ANALYSIS

SECOND EDITION

Co-ordinated By
Water Quality Section
Laboratory Services Branch

July, 1977



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A GUIDE TO THE COLLECTION AND SUBMISSION OF SAMPLES FOR LABORATORY ANALYSIS

INTRODUCTION

Sample collection is the first and commonly most critical stage in the step by step procedure used to determine a substance or group of substances in the environment. From the standpoint of data interpretation, it is normally assumed that a representative sample has been taken. If the sample is not, in fact, representative, then this should be noted to avoid erroneous data interpretation. Similarly, once the sample has been collected, improper use of preservation techniques to stabilize the sample during transportation will lead to useless results.

In general, the sampler's aim must be to collect a representative sample from a known position (location) and transfer it to the laboratory with a minimal change in chemical composition of the parameter of interest. It is of little value to make an accurate analysis of an incorrectly collected sample.

IT CANNOT BE EMPHASIZED TOO STRONGLY THAT THE SAMPLER PLAYS A KEY ROLE IN ENSURING THAT THE DATA OBTAINED ACCURATELY REFLECTS THE FIELD SITUATION BEING ASSESSED.

ONTARIO MINISTRY OF THE ENVIRONMENT - LABORATORY SERVICES BRANCH

The various laboratories of the Laboratory Services Branch are equipped to perform a large number of chemical and microbiological analyses on domestic water supplies, surface waters, ground waters, and domestic and industrial wastes. Fish, vegetation, soil samples, hi-vol filters, precipitation and snow samples are also analyzed by these laboratories. Special analyses required for research studies and unusual pollution problems can also be provided.

Decentralization of the Ontario Ministry of Environment has resulted in the designation of six regions with boundaries as given in Figure 1. Three of these, the Northwestern, Southwestern, and Southeastern, have regional laboratories located at Thunder Bay, London and Kingston respectively. Samples collected within these regions are analyzed at the appropriate regional laboratory when analytical capability is available. When such capability is not available, these samples plus all those from the other three

regions are analyzed at the Toronto Laboratory. The chemical tests performed by each regional laboratory as well as the six sectional laboratories in Toronto, are outlined in Table I. Those tests which are performed by the Laboratory Services Branch and are unique to Air Quality Assessment are given in Table V. Field samplers should ensure that a laboratory is capable of analyzing the parameter (s) in question *before* shipping. Mobile laboratories and field stations are often provided to perform a limited number of tests in conjunction with major surveys or studies.

While the six sections of the Toronto Laboratory (Water Quality, Air Quality, Organic Trace Contaminants, Pesticides, Physical Methods, and Microbiology) are located at the main facility on Resources Road, parts of the Air Quality Section are found at 880 Bay Street, 3rd floor. By 1978, however, all these laboratory services will have been consolidated at Resources Road.

A. SAMPLE COLLECTION FOR WATER QUALITY ASSESSMENT

A - I. SAMPLE COLLECTION FOR CHEMICAL ANALYSIS

1. GENERAL CONSIDERATIONS

The method of sample collection in the field is the responsibility of the individual involved. The following points should be noted:

- a) The sample must be truly representative of the whole.
- b) All possible sources of sample contamination (sampling devices, motor exhausts, disturbing of bottom sediments, use of inappropriate containers, etc.) should be eliminated or reduced to a minimal level.
- c) Since sample composition will change with time, rapid transportation to the laboratory is desirable. For some parameters, use of a preservative is recommended. Theoretically, this fixes the concentration of the parameter of interest and reduces the need for rapid transport and analysis (See Section A-III). However, in practice this only delays the perishability of the parameter and the sample should still be transported as quickly as possible.

- d) For samples which do not have a preservative already in the collection bottle, rinsing *both* the bottle *and* cap with sample (two or three times) is strongly recommended. This procedure, while reducing any contamination that may be present, also tends to equilibrate the sample with the container walls and hence "container effects" (leaching, adsorption, etc.) are minimized.

2. SAMPLE CONTAINERS

In general, surface water samples are collected in 1 litre glass bottles, but some analyses require plastic containers. Table II summarizes the bottle type recommended for each parameter. Special studies may specify a certain container type and prior to collecting samples for such studies, the sampler should check on this requirement.

Sludge samples are collected in wide mouth glass bottles and *never* filled more than half way. The extra space is required as an expansion zone for gaseous products that may be formed. Failure to handle these samples in this manner may result in container explosion during transit or at the laboratory. Overfilled sludge samples are discarded without analysis.

For trace level heavy metal analysis on surface and domestic waters, special acid washed plastic containers are required. Standard glass containers with non-metal cap liners should be used for all other routine water samples requiring metal determinations.

Note that samples submitted for physical testing such as particle identification (See Section D-11 or microbiological analysis are generally unsuitable for chemical analysis and vice versa. An appropriate separate bottle should be submitted for each of these test types when more than one is required.

3. PRESERVATION TECHNIQUES

The function of a preservative is to stabilize the parameter of interest so that changes in composition during transit and prior to analysis are minimized. Several different preservation methods are recommended and these are outlined by parameter in Table II.

Preservation techniques usually involve the addition of a chemical which "ties up" the parameter in a form which is unaffected by sample ageing or else provides conditions unsuitable for any further reaction to occur. In some cases, refrigeration or freezing to reduce reaction rates provides the best preservation, particularly for those parameters which have a direct biological relationship (i.e. with respect to growth or decline, for example, nutrients).

The sampler should be aware that the use of the recommended preservative for one parameter may negate the possible analysis of another. For example, heavy metal samples preserved with nitric acid are unsuitable for phosphate analysis. It is the sampler's responsibility to determine from Table II whether use of a certain preservative will eliminate the possibility of analysis of another requested parameter, and provide suitable duplicate samples to avoid the problem. If in doubt, consultation with laboratory staff is advised. Each duplicate should have the preservative used clearly marked on the bottle label.

4. SAMPLE VOLUME

The analytical methods used to determine parameter concentrations require a certain minimum volume of sample in each case, as outlined in Table II. The field sampler is expected to calculate the total volume of sample required by summing the specified individual volumes (Table II) for all the analyses requested and to submit the appropriate quantity. In addition, samplers are asked to submit at least *six ounces* of sample in excess of their original total estimate to allow for possible repeat analysis. Failure to provide sufficient sample volume will normally result in "Sample Exhausted" being marked on analysis report sheets.

In most cases, the volume required for analysis depends on parameter concentration, with "clean" samples (i.e. low concentrations) needing the largest amounts. Domestic water supplies, well waters, and unpolluted surface waters fall in this category. Tests for these sample types require the largest practical volume in order to provide a sufficient quantity of the substance of interest for reliable detection. Samples of high concentration (effluents, sewages, etc.) require a much smaller amount, and even a dilution may be employed.

In certain cases where the sampler is unable to obtain sufficient sample volume or when re-sampling is impossible, analysis may still be obtained if special care and analytical

techniques are used in the laboratory. This can only be achieved *after* consultation with laboratory personnel has been initiated by the sampler and *before* sample submission.

A number of tests may be performed simultaneously on a single aliquot, consequently certain commonly requested combinations of parameters may require a smaller volume than expected. These are discussed on page 9.

5. SAMPLING METHODOLOGY

The sampler should be aware of how the particular details of his procedure (geographic location, time of day, method of obtaining the aliquot, etc.) may bias the results which are eventually obtained.

Care should always be taken to minimize sample cross-contamination by carefully rinsing (with sample) all materials used in collecting the aliquot which is sent to the laboratory. These precautions are particularly important for low concentration parameters.

6. FIELD RECORDS

It is in the sampler's own interest to keep complete records of his sample collection activity not only from the standpoint of date, sample number, location, description, etc., but also with regard to unusual features which may be extremely useful in interpreting the analytical data. This information may also prove invaluable in the event of sample loss, misnumbering of sample bottles or report sheets, etc.

A - II. SAMPLE COLLECTION FOR MICROBIOLOGICAL TESTING

1. GENERAL CONSIDERATIONS

It is the responsibility of the sampler to use aseptic techniques when handling the sterile bottles used for microbiological sample collection. Failure to do so will result in sample contamination and meaningless results. It is recommended that the techniques described below be closely followed in order to obtain reliable data.

2. SAMPLE CONTAINERS

Pre-sterilized 8 oz bottles with blue labels are usually adequate for the routine sampling of water from distribution systems, surface water, well water, etc., provided the waters have not been chlorinated. Chlorinated waters must be sampled in pre-sterilized 8 oz bottles containing sodium thiosulphate (red label). Samples collected for special "nuisance" organism analyses should be collected in regular (blue label) bottles.

Samples collected at depth, are taken using sterile sampling bulbs obtained from the Microbiology Section.

3. PRESERVATION TECHNIQUES

Sodium thiosulphate is used to neutralize the disinfecting properties of chlorine thereby preserving the existing microbial population at the time of sampling. This preservative is already present in the red labelled sample bottles. Keep samples cool, preferably through refrigeration or ice, and away from light during transportation to the laboratory. Frozen samples will not be accepted.

4. SAMPLE VOLUME

In general, one bottle or bulb per sample provides sufficient volume for standard analysis. If, however, the bacteria levels expected are very low or extra parameters are being requested, then additional samples may be required. Consultation with Microbiology Staff is advisable in such cases.

5. SAMPLING METHODS

Sterile sampling bottles are available through Central Stores in Toronto, and regional laboratories. For special studies, alternate bottles are obtainable through Microbiology Staff on consultation. Samplers should check to see if the plastic seal on each container is intact before sampling. Containers with loose or cracked seals should *not* be used. All samples should be collected early in the week and shipped to the appropriate laboratory. During spring, summer and fall, samples should be packed in ice to minimize biological activity. In winter,

samples should be packed in insulating material to prevent freezing while still keeping them cold. Immediate delivery to the laboratory is *essential*. Analysis within six hours is preferable, but should not exceed twenty four hours.

Strict adherence to the following sampling procedures is recommended:

a) SURFACE SAMPLES

Clamp the bottle onto a sampling pole before removing the cap. Touch only the outer surface of the cap when opening the bottle. The inner lip and liner must not come in contact with anything except the atmosphere. If the inner surfaces of the cap or bottle lip are accidentally touched, the sample has been contaminated and should not be submitted. The recommended procedure is to hold the cap with your fingertips until the sample has been taken. The cap must not be set down somewhere while the sample is being taken as this will result in contamination.

Surface sampling is accomplished by quickly lowering the sample bottle into the water approximately *one meter* below the surface with the mouth facing into the current. When sampling near shore, care should be taken to get a sample uncontaminated with sediment. The bottle is then removed from the water, the level adjusted to the top of the label, immediately capped, and then unclamped from the sampling pole. Samples must be collected using this prescribed technique. The use of a dipper or other sampling device will result in contamination.

b) DEPTH SAMPLES

Depth samples are taken using sterile sampling bulbs. Bulbs should be used as quickly as possible: if not used, they should be returned to Microbiology Staff within a maximum of two weeks, otherwise, the rubber will crack and the bulb will not open. The same care that is used with sampling bottles must be used in the handling of bulbs. The glass plugs supplied have been sterilized within a cellophane envelope and must not come in contact with any contaminated surfaces when they are being removed from the cellophane envelope. If, for some reason, the sampler should run out of glass rods, he may dip the metal plug into alcohol and flame it. After flaming, the plug is immediately inserted into the bulb, taking

the usual precautions when handling sterile equipment. The use of the metal plug is discouraged and it should only be used in rare instances when the sample could not possibly be obtained at a later time in the correct manner.

c) TAP SAMPLES

Samples from taps must be taken only after aerators, screens, hoses, etc., have been removed. Prior to sampling from a tap, the water should be allowed to run at full flow for approximately *two minutes*. The strong flow will clean out residual contamination around the orifice of the tap thus ensuring a more representative sample. The water pressure may then be reduced to permit taking the sample without excessive splashing which could result in contamination of the sample.

Fill the bottle to the top of the label being certain that the mouth of the bottle does not come in contact with the tap or any contaminated surface. The cap must also be handled aseptically as described previously.

d) SAMPLE DUPLICATION

When a duplicate sample is being taken, it should be obtained at the same time as the first sample. This can be achieved for surface samples by clamping two bottles on a sampling pole, and for depth samples by placing two bulb samples on the sampling line in a "piggy-back" fashion.

A - III. CHEMICAL AND PHYSICAL FIELD ANALYSIS

The perishability of some parameters for which no chemical preservative is suitable necessitates field measurement. In the case of major field studies, a field laboratory facility for this purpose may be warranted. For example, such parameters as dissolved oxygen, dissolved carbon dioxide, free chlorine, chloramines, hydrogen sulphide, temperature and pH are extremely perishable so *on-site* analysis is recommended. Temperature, pH and dissolved oxygen are conveniently measured using electrode sensors (and/or Winkler titration for dissolved O₂) while dissolved carbon dioxide, free chlorine, and chloramines require more complicated analytical techniques. Prior consultation with the Laboratory Services Branch, Water Quality Staff, is recommended in these cases.

A - IV. SAMPLING BY PARAMETER

Specific information with regard to recommended sample bottle (*in order of preference*), preservation techniques (*in order of preference*), and volume required for each parameter, is given in Table II. Simultaneous analysis of certain parameters on a single aliquot allows a reduction in the volume required as follows:

- a) Both total phosphorus and Kjeldahl nitrogen can be determined on a single 75 mL (3 oz) aliquot.
- b) Nitrate nitrogen, nitrite nitrogen, ammonia nitrogen, and filtered reactive orthophosphate can be determined on a single 75 mL (3 oz) aliquot.
- c) Both sodium and potassium can be determined on a single 40 mL (2 oz) aliquot.
- d) Aluminum, copper, cadmium, cobalt, chromium, iron, lithium, molybdenum, manganese, nickel, silver, strontium and zinc can be determined on a single 400 mL (14 oz) aliquot. For very low levels, preconcentration necessitates that one full container (900 mL) be submitted for metals.

A - V. PARAMETER GROUPINGS

Although the laboratories have analytical capabilities for a wide diversity of water quality parameters, certain compatible groupings are requested with a consistently high frequency. Such group requests are usually associated with routine monitoring programs and/or specific projects. It is the nature of the groupings to allow analysis of all the specified parameters on a single or sometimes duplicate sample bottle. The six most common groupings are given in Table III.

Requests for one of these groupings should be made only when ALL the parameters are required. Specific environmental problems usually require specific analysis be performed and, therefore, use of these groups is of little value. Large projects and studies may find it advantageous to use a grouping different from those in Table III and these may be established *after* appropriate consultation with the proper laboratory personnel.

- a) The general submission number (only) if and when this is required.
- b) A sample (sender's) number.
- c) Some other identification (e.g. "Rock Inlet").
- d) Presence of any *chemical* preservative added; all others will be kept refrigerated or frozen (i.e. as received) until time of analysis.
- e) When appropriate, indication of a single specific analysis required for that one sample bottle; i.e. when the sample has been preserved for resins and fatty acids analysis, it should be labelled "For Resin Acids", or when submitted for preconcentration and heavy metal analysis, it should be labelled "For Preconcentration".

2. SUBMISSION FORMS

Submission forms must accompany all samples and should include the following information, completed in pen, preferably black.

- a) The required analytical parameters listed in the designated space on the form. This listing should always be present *including* the occasions when a parameter group is specified. Samples cannot be accepted with such requests as "chemical analysis" or "all the metals". Specific parameter identification is necessary and in some cases (i.e. pesticides), the specific class or compound is required. If there is some doubt concerning which analysis to request, a brief description of the general problem or reason for sampling will enable the laboratory staff to select the appropriate tests.

- b) The sender's number corresponding to the number marked on the bottle.
- c) The other sample identification provided on the bottle.
- d) The sampler's name.
- e) The name, address *and* phone number of the person to whom the results are to be reported.
- f) Sampling date.
- g) Program or study under which the sample was collected. (Note region or head office branch).

3. GENERAL

If a group of related samples is submitted, a map of the area or the relative location of waste inputs is very helpful to the laboratory staff. Please number the samples in a logical order, e.g. downstream in a river. All analyses are routinely screened for anomalous results before they are released. Suspicious results might be improperly rejected whereas some details as to the location would confirm their validity. When a sample is sent to the laboratory, a description of known constituents should be included on the submission sheet, particularly an unusual one such as industrial waste contaminant. Interferences (reactions which produce false analytical results) can be eliminated by pretreatment if the analyst is forewarned. Samples known to contain cyanide, arsenic, mercury or other toxic materials should be clearly marked (warning label with substance identification) for the protection of laboratory personnel.

When submitting samples for analysis of organic contaminants, be as specific as possible about the types of compounds to be determined, and also, when a specific source of contamination is suspected, send samples of the source material for comparison. In all cases, the use of glass bottles with foil or teflon lined caps is a necessity for organic samples. Sampling foams is particularly troublesome. This may be best achieved by sampling just the foam with a pomade jar, then breaking the emulsion, and repeating the process until sufficient volume is obtained (usually at least 10 times).

Identification of unknown contaminants is very time consuming. Samples should be as large as possible to allow a wide range of exploratory tests. The sampler should indicate whether

qualitative or quantitative results are required. Any available information concerning the sampling point, possible contaminants, and industries implicated is extremely important for such samples. Organic compounds not readily identified by the other units in the OTC section, may be submitted to the Mass Spectrometry unit for analysis.

Samples which are not homogeneous present analytical difficulties because it is virtually impossible to take a representative aliquot. If the sampler is only interested in one phase (aqueous, solid or immiscible organic), he should label the submission form accordingly. Otherwise the laboratory will consider the whole sample, and take aliquots of the mixture.

Samples sent to the Central Laboratory, Toronto, could be analyzed by as many as six different sectional laboratories; the distribution of analyses among these sections is shown in Table I. Sample processing is much more efficient if the sampler submits separate forms and separate sample containers if testing is to take place in more than one of these sectional laboratories. Your co-operation in this respect will be appreciated by everyone involved.

4. SAMPLE CONTAINER REQUISITION AND SHIPPING PROCEDURES

Sample containers may be requisitioned according to need using the information provided in Table IV. It should be noted that 32 oz or 20 oz glass bottles are no longer available.

Certain projects or studies may require the use of special container types, and appropriate enquiry should be made prior to requisition.

CN Express and CP Express provide the fastest and most reliable service for the shipment of environmental water samples in Ontario. Air express, parcel post, bus companies and other services discourage the shipment of water samples because of the damage caused to other shipments when breakage occurs.

Contract numbers are important as they provide the only means for tracing a lost shipment. Every shipment is assigned a contract number at the Express Depot, but it is generally up to the sampler to attach this contract number to each carton of his shipment. Identification stickers are provided by the Express companies upon request. Samplers are urged to keep a record of all their contract numbers.

The following addresses should be used when shipping samples to the various laboratories:

a) CENTRAL REGION - MAIN TORONTO LABORATORY

Ontario Ministry of the Environment,
Central Stores,
Resources Road,
Highway 401 and Islington,
Toronto, Ontario.

b) SOUTHWESTERN REGION - LONDON LABORATORY

Ontario Ministry of the Environment,
Southwestern Regional Laboratory,
985 Adelaide Street South,
London, Ontario.

c) NORTHWESTERN REGION - THUNDER BAY LABORATORY

Ontario Ministry of the Environment,
Thunder Bay Regional Laboratory,
421 James Street South,
Thunder Bay, Ontario

d) SOUTHEASTERN REGION - KINGSTON LABORATORY

Ontario Ministry of the Environment,
Southeastern Regional Laboratory,
133 Dalton Street,
Kingston, Ontario

Further enquiries regarding container requisitions, shipping, etc., should be directed to Central Stores in Toronto (Telephone 248-7134).

A - VII. SAMPLING FOR ASBESTIFORM MINERAL FIBRE

The analytical technique for asbestos determination involves a time consuming electron microscopic inspection of the sample in the Physical Methods Section, Resources Road, Toronto. The extreme care and time required for this analysis makes it a very costly test, and very long sample back-logs are common. For these reasons, sampler discretion regarding submission of asbestos samples is requested. *No sample should be submitted without previous consultation with Physical Methods personnel.*

Water samples should be collected in a 1 litre plastic bottle. As a rule, only new bottles should be used. On request, pre-cleaned bottles will be supplied by the laboratory. The usual precautions of multiple bottle rinsing, rapid transport to the laboratory etc., are of particular importance for the collection of asbestiform mineral fibre samples. Samples should be sent to the Physical Methods Section, MOE, Resources Road, accompanied with the sample submission form.

A - VIII. SNOW SAMPLING

Snow sampling should be carried out using sample collectors designed to catch it as it falls. In some cases, however, collection of the snow as it lies on the ground may be necessary or desirable. This should be accomplished by shovelling the snow into a large pre-cleaned plastic bag or bucket and then thawing at room temperature prior to quantitative transfer into glass or plastic bottles. Generally, a *half-square meter* collected to a depth of 20 cm will provide sufficient sample volume.

Care should be taken to:

- a) not include any soil or vegetation beneath the snow
- b) always use a plastic shovel (rather than metallic)
- c) vigorously mix the melted sample before transferring to the bottles

Once in the bottles, the sample should be handled in the same fashion as other water samples, i.e. with regard to storage, preservation, shipping, etc.

A - IX. ENQUIRIES

Attention is drawn to the "Outlines of Analytical Methods" available from the Water Quality Section, Resources Road, Toronto, for more specific information with respect to parameter descriptions, analytical methods, sampling restrictions.

People to whom enquiries must be addressed by telephone are listed in Appendix I.

All samples received by the Laboratory Branch are assigned numerical codes according to sample type. When the analyses are completed, the results are entered onto the original submission sheets, checked by the scientist-in-charge, and sent for typing. All original submission sheets are retained in the sample reception files. Laboratory staff are prepared to answer questions regarding the receipt and progress of samples but require the following information:

- a) Municipality, Township, or body of water in which the sample source is located
- b) Name of person etc. to whom the analytical report is to be submitted
- c) Name of program or study
- d) Sampling date and estimated day of arrival at the laboratory
- e) Location codes or other sample identification number. Laboratory numbers are preferred, if known
- f) Type of sample (e.g. water, river, sewage, industrial wastes, Great Lakes, etc.)

B. SAMPLE COLLECTION FOR AIR QUALITY ASSESSMENT

Assessing air quality problems usually requires specialized techniques, and prior consultation with Air Quality Laboratory personnel is recommended. Before instituting a new survey, the laboratory staff should be consulted regarding sampling locations, frequency, and analysis requirements. The laboratory which performs most of the analysis in this regard is located at 880 Bay Street, Toronto; however, by 1978 all facilities will be centralized at the Resources Road laboratory. Testing capabilities have been outlined in Table I; those parameters unique to Air Quality Assessment are given in Table V and are discussed at length below.

B - I . DUSTFALL SAMPLING

A clean sealed polyethylene dustfall collector jar (*12" tall X 6" diameter*), identified by Station number, is attached to a suitable supporting bracket, uncovered, and allowed to collect settleable particulate material over a one month period. Collectors are located to provide dustfall samples that are representative of the area being studied.

Each collector should have a clear field of exposure, free from interferences such as buildings or other high objects or structures. Accessibility and security are other considerations in site selection.

The top of the container should be a minimum of *8* and a maximum of *50 feet* above the ground and at least *four feet* above any other surface in the vicinity. Attachment to hydro poles is a common method of support.

During summer, an aqueous solution containing *1 mg/l* of CuSO_4 may be added as an algal and fungal inhibitor provided that these substances will not affect the desired analysis. A dustfall collector containing *two litres* of the above solution tends to prevent loss of particulate matter by the action of wind currents. The use of "wet" collectors is not universal, and consultation with laboratory staff is recommended prior to their use.

It is important to establish the down-wind direction from the source being investigated and position the dustfall collector accordingly. After one exposure period the collector should be removed, capped, and *taken* to the laboratory for analysis. Since the collector must be kept in an upright position, shipping by CN, CP etc., is not feasible.

It is very important that a record of station number and installation and removal dates accompany the collector since this information is necessary to calculate the results.

B - II. "HI-VOL" FILTER SAMPLING

The collection of suspended particulate material involves filtration of air through an 8" X 10" glass fibre filter using a vacuum pump capable of drawing at least 40 ft.³/min. The normal sampling period is 24 hours. A complete description of the Hi-Vol sampling device and procedure may be obtained from the ASTM publication, Gaseous Fuels; Coal and Coke; Atmospheric Analysis, Part 26, November 1975.

The sampler consists of a face plate, gasket and retaining ring, a filter adapter assembly, and a vacuum pump. The sampler is mounted vertically within a protective shelter. With the pump drawing 60 ft.³/min., the louvered shelter will only allow suspended particulates up to approximately 100 microns (aerodynamic diameter) to reach the filter. Pre-weighed and coded glass fibre filters and protective envelopes are available from the Air Quality Laboratory for use with these samplers. The filter must be carefully installed (rough side upwards) on the sampler, and the coded number recorded on the envelope. Ripped or punctured filters must be discarded. If difficulty is encountered due to wind, it may help to switch on the motor, thus holding down the filter while it is being secured by the frame. The collection surface of the filter should not be touched at any time. Once the sampler has been prepared, the motor should be switched on and the air flow measured using the orifice manometer and the reading recorded on the envelope together with the pre-set time for start up.

The operator should then shut down the pump and reset the timer. The pump will then automatically start up and usually runs twenty-four hours (midnight to midnight). Once the sample has been collected, and before the filter is removed, the pump should be momentarily switched on and the final air-flow reading recorded as well as the day on which the filter was exposed. After carefully removing the filter, fold it in half along the 8 inch width, particulate side inwards and place in the corresponding envelope. Any comments peculiar to the sampling conditions should be noted. This is important for data evaluation.

The filter should be mailed to Air Resources Branch, 880 Bay Street, (4th Floor), Toronto, for calculation of the air volume. The filter is then forwarded to the Air Quality Section for analysis. Hi-Vol filters for the Northwestern Region are obtained from and sent to the Regional Laboratory in Thunder Bay.

Analysis of non-volatile contaminants, such as benzo-a-pyrene (BaP), benzo-k-fluoranthene (BkF), polycyclic-aromatic-hydrocarbons (PAH), and benzene soluble organics, may be performed on sub-samples from the Hi-Vol filters. Field sampling instructions in this regard are the same as above. These filters should be kept protected in envelopes and not be exposed to heat or sunlight.

Certain inorganic tests are incompatible with the glass fibre filters normally used. These include: Al, Ba, B, Ca, Hg, Na, K, Si, and F. For these elements, except mercury, polystyrene (Delbag) filters are recommended and are available from the Air Quality Section. Note that total suspended particulates and organic contaminants cannot be determined on Delbag filters. For Hg sampling, see Section A-VIII.

In summary, the filter envelope must contain the following information:

- a) Station number (i.e. sampling location)
- b) Hi-Vol instrument number, date and time of exposure
- c) Filter number
- d) Operator
- e) Flow readings at start-up and shut-down
- f) Comments regarding incidents peculiar to the sampling period.

B - III. SULPHATION AND FLUORIDATION CANDLES AND PLATES

Sulphation and fluoridation rates are measured using the appropriate "candles" or "plates". These devices may be obtained from the Air Quality Section. The candle or plate should be removed from its protective cover and placed over the peg inside the candle cage or in the plate holder. After approximately 1 month's exposure the device should be replaced in its protective container and returned to the Laboratory. A complete description of sampling considerations for sulphation rate can be found in the ASTM "Gaseous Fuels; Coal and Coke; Atmospheric Analysis", Part 26, November 1975.

Protective shelters are provided and installed by the regional staff. The stations should be located between 8 and 15 feet off the ground, and should be isolated from any obvious local interferences. Normal exposure time is *thirty days*. The exposed candle or plate should be carefully replaced in its protective cover, placed in its shipping container and sent to the Air Quality Section. Proper sealing of the candle or plate is important to prevent further atmospheric reaction occurring during transit. Regional staff should take care not to touch the reactive surface of the candle or plate at any time. The duration of exposure must be recorded and submitted with the candle or plate. Plates are recommended for any new surveys.

B - IV. VEGETATION AND SOIL SAMPLING

1. GENERAL

The Phytotoxicology Section, Air Resources Branch (880 Bay Street), is responsible for the investigation of all complaints concerning suspected air pollution damage to vegetation or contamination of soil and the establishment of all vegetation and soil assessment surveys in the vicinity of proposed or existing industrial emission sources. The exception is in the NE and NW Regions, where the work is performed by the Technical Support Sections, with the assistance as required from Phytotoxicology personnel.

2. TYPES OF INVESTIGATIONS

a) ASSESSMENT SURVEYS

These surveys are conducted to document endemic conditions prior to the establishment of emission sources, to define the current state of air emissions from existing sources, and/or to monitor source compliance with Ministerial orders. Normally, a sampling grid is constructed, centred on the source and samples are taken from established stations located at increasing distance along radii from the source to the limits of suspected contamination. Consideration is given to the location of air quality monitoring instruments and meteorological parameters such as prevailing wind direction.

b) COMPLAINT INVESTIGATIONS

Samples may also be taken to evaluate situations where extensive damage to vegetation has been observed. Cases of this nature will usually be drawn to the Ministry's attention through complaints by individual citizens. All complaints of this nature should be referred to the Phytotoxicology Section. They will be investigated and reported to the individual originating the complaint and to the source of the contaminant.

3. SAMPLING PROCEDURES

To ensure a correct interpretation of analytical data, all samples that are to be compared must be carefully matched with regard to plant species, age or maturity of leaf tissues, age of tree or shrub, and position of sample on tree or shrub. Usually, foliage is collected from the side of the tree or shrub facing the presumed source of air pollution but, occasionally, a second sample may be taken from the side opposite from the source. Samples are taken by trimming outside growth from ground level up to 20 ft or more and collecting all leaves to provide a composite sample of 500 to 1,000 grams of fresh material.

Current practice is to collect three samples from each sampling location (triplicate sampling). Samples are placed into perforated polyethylene bags and are transferred to refrigerated storage as soon as possible for processing in the Phytotoxicology laboratory. Forage samples (grass) are collected by cutting the terminal 25 cm (10") of stems and blades over the representative area to be sampled, at 10-step intervals. Dried flower heads and stalks are discarded and no root material whatsoever is included. The different forage species included in the sample are identified and are representative of the population of the species in the field.

Any sample contaminated by roadside dust should be noted in the accompanying request form.

Soil samples are normally collected in conjunction with vegetation samples as an aid to differentiate between current and past emission situations. Occasionally, soil samples will be collected to establish background conditions.

Soil is collected with a 2 cm (3/4") diameter stainless steel tube. A minimum of 10 cores is taken from the sampling site. Also, all soil samples are collected in triplicate (i.e. minimum

3 X 10 cores) and the collection form is completed to comprehensively describe the texture of the soil and the over-all sampling site. Each core must be separated into fractional depths of 0 - 5 cm, 5 - 10 cm and 10 - 15 cm, and each level is placed in an appropriately labelled plastic bag for shipping.

Ideally, soil should be sampled from an entirely undisturbed or sodded area and contaminated situations should be as closely matched as possible with conditions existing immediately outside of the area.

4. SAMPLE STABILIZATION

All vegetation samples as collected, are potentially unstable, and will decompose unless properly handled. Vegetation samples can be preserved for a few weeks under refrigeration; when dried at 80°C for 30 hours in forced draft oven, they become almost permanently stable.

5. SAMPLE IDENTIFICATION

Collection of vegetation and soil samples is accompanied by the completion of a prenumbered PS2 Form (Phytotoxicology Field Sample Collection Form) which will later provide all the necessary information required for interpretation of the test results. The lower portion of the form is detachable and is placed in with the sample for identification. Normally samples are "double-bagged" with the numbered field sample enclosure slip placed between the outer and inner bags.

B - V. ORGANIC CONTAMINANT SAMPLING

Sampling for the analysis of organic contaminants commonly require special methods and equipment, and prior consultation with the laboratory is essential in all cases.

Techniques used for sampling of these components are discussed in Sections II (Hi-Vol) and VII (Special Techniques).

B - VI. SAMPLING FOR ASBESTIFORM MINERAL FIBRES

The analytical technique for the determination for asbestiform fibres in air involves a time consuming electron microscopic examination of the processed samples. The expertise, time and instrumentation required for this analysis make it a very costly test. For these reasons, sampler discretion regarding submission of samples is requested. Every attempt should be made to preserve the integrity of the sample.

Asbestiform minerals insuspended air particulate are collected on a 0.4 μ m pore size nucleopore filter using a modified Hi-Vol sampler. The modification consists of installing a flange with a 3/4" dia. opening on the air exit of the sampler. This opening acts as a limiting orifice and brings the air flow rate into a suitable measurement range. It is recommended that the Hi-Vol sampler be equipped with a transducer and an air flow rate recorder. The sampler must be recalibrated after the modifications have been performed. Procedures for calibration may be obtained from the laboratory or from the Technical Services Group, Central Region, 880 Bay Street, 1st Floor, Telephone: 965-2129.

It is very difficult to change the filter in the field and *pre-installation* of the filter in the Hi-Vol Cassette *inside an enclosed area* is recommended. The entire cassette assembly is then attached to the air sampler. Removal of the filter should be performed in like manner.

After exposure, the filter is removed from the cassette, placed on the 8m X 10m separator sheet supplied with the filter and both then folded along the 8 in. width. The folded filter and separator are placed within a glassine envelope and mailed to the laboratory in the usual kraft paper Hi-Vol envelope, together with all pertinent sampling data. Samples requiring asbestos analysis should be mailed to the Physical Methods Section, Resources Road, Toronto.

B - VII. NON-ROUTINE TECHNIQUES

The following are techniques which may be used in special circumstances, after discussion with laboratory staff. The sampling of volatile organic contaminants are covered by some of these techniques.

1. LOW VOLUME SAMPLING

"Low volume" techniques include sampling with impingers, adsorption tubes, and filters. Samples

for the analysis of volatile organic components such as vinyl chloride, peroxy-acetyl-nitrate (PAN), volatile aliphatic and aromatic hydrocarbons may be collected by passing 100 to 1,000 ml of air per minute through a specially prepared tube containing activated charcoal, Chromosorb, or another suitable adsorbent. The normal sampling period is 2 - 4 hours. The tubes are available from the Organic Trace Contaminant Section. The sample, once collected, must be refrigerated and kept in the dark. The sample label attached to the tube must have the following information marked on it:

- a) Date and location
- b) Pump time on and off
- c) Air flow rate at the start and finish
- d) Wind speed, direction, and temperature

Samples should be shipped (mailed) to the Organic Trace Contaminant Section, Resources Road, Toronto.

2. GRAB SAMPLES

An alternative way of sampling for volatile contaminants is by collecting a "grab" sample in Tedlar bags, aluminized Mylar bags, evacuated glass and metal containers, etc. A grab sample is taken by pumping air into the bag or filling an evacuated container with air. This sampling method may be applicable in cases of odour problems.

An important consideration is that the contaminant does not react with or adsorb on the material of the container.

3. CASCADE IMPACTORS

Impactor type samplers capable of separating particulate matter into size ranges according to their aerodynamic size are available. The cascade impactor separates particulate matter within the respirable range ($.3 - 10 \mu\text{m}$).

The sampler may be used for differentiating between sources of pollution. For example, lead emitted from automotive sources is found in the submicron fraction, while lead emitted from certain industrial operations as particles is deposited in the larger than one micron fractions.

The available impactors are used in association with Hi-Vol samplers. A problem specific to the cascade impactor is that the jets become clogged with dirt and require frequent cleaning to maintain its calibrated flow rate.

4. DETECTION TUBES

By observing the length of discolouration produced in a solid absorbent of a specific tube through which a known small volume of air is drawn, the approximate concentration of a pollutant can be estimated. This method is a rapid, semi-quantitative procedure for measuring high levels of gaseous pollutants (SO_2 , CO, H_2S) in the field.

5. CONTINUOUS AIR MONITORS

Sulphur dioxide, oxides of nitrogen, ozone, oxidant carbon monoxide, and reactive hydrocarbons are the pollutants that are regularly monitored as part of the air monitoring program in Ontario. Each region is responsible for maintaining and calibrating their own monitors.

Instruments have also been developed for other pollutants such as hydrogen sulphide, fluorides, vinyl chloride and mercury.

Sequential filtration samplers are also used to measure the soiling property of ambient air in co-efficient of haze units. The air sample is drawn through a paper filter tape on which the particulate matter is deposited. The period of sampling is variable generally being one or two hours. The deposit may be removed for elemental analysis.

Continuous air monitors are the best means of sampling and analyzing gaseous pollutants because the effects of handling and perishability are minimized by direct introduction of the sample into the instrument. This technique is expensive, requiring a heated shelter equipped with power and trained technicians to calibrate and maintain instruments on a regular and frequent basis.

6. STACK SAMPLING

Stack samples can be obtained by inserting probes into a vent through which gaseous or particulate emissions pass to the atmosphere. Emission rates can be calculated from analysis of samples. Rigid procedures must be followed in stack sampling to ensure representative samples are taken. Most of this sampling is carried out by experienced outside agencies. Analytical work on stack samples has been carried out in conjunction with investigations on special industrial source emissions.

C. SAMPLE COLLECTION FOR THE ANALYSIS OF SEDIMENT, SOIL AND BIOMATERIALS

C - 1. COLLECTION OF SOIL, SEDIMENT, AND VEGETATION SAMPLES FOR INORGANIC CONTAMINANTS ANALYSIS

1. ANALYTICAL TESTING CAPABILITIES - SOILS AND SEDIMENT LABORATORY, AIR QUALITY SECTION

In addition to the tests listed in Table I as performed by the Air Quality Section, the Soils and Sediment Laboratory carries out the sample preparation on solid samples for a number of tests performed by other sections.

These are given in Table VI along with the required sample size and method of preparation.

2. GENERAL SAMPLING CONSIDERATIONS

- a) Where possible, composite sampling will result in a more representative sample than a single grab.
- b) All possible sources of sample contamination should be reduced to a minimal level.
- c) Chemical preservatives are generally not applicable to samples of this type.
- d) Preparation of sediment, soil and vegetation for chemical analysis generally takes longer than for water or effluent, and as a result, samples requiring immediate attention should be so marked *after* prior consultation with laboratory personnel.
- e) Samplers should be aware of pertinent information regarding submission procedures as outlined in Section A - VI.

3. SAMPLE CONTAINERS

Any clean glass or plastic container is acceptable for sediment, soils or vegetation samples. While not recommended, paper bags can be used for "dry" soil samples where contamination is not anticipated to affect the analysis (e.g. particle size analysis). In general, wide mouth 2, 4, 8, 16 or 32 oz glass or plastic containers are the most suitable, depending upon sample size. When a number of samples are submitted as a series, uniformity of sample container size is recommended for shipping, handling and storage convenience. Plastic bags (Twirl-top) are usually adequate for dried vegetation samples.

4. PRESERVATION TECHNIQUES

Chemical preservation techniques are generally not applicable to solid type samples. In the short term, storage at 4°C or freezing will minimize the transformation of species, particularly if a soluble for "available" parameter is desired.

Drying at 80 - 100°C as a preservation technique is recommended except for those samples requiring analysis of potentially volatile parameters. In practice, a portion of each sample received in the laboratory is oven dried at 110°C after which most chemical tests are performed. In general, dried samples are indefinitely stable.

Air or oven drying of vegetation samples before submission to the laboratory is recommended and where this is not possible, the laboratory must be notified so that the samples can be dried without delay. This is particularly important since decaying plant material could significantly alter nutrient values.

5. SAMPLE SIZE

The field sampling personnel must be aware of the general non-homogeneity of solid samples and thus the minimum sample size should reflect this consideration. As a general rule, however, a sample which will yield 10 - 25 g. of dry material will be sufficient for all the routine chemical analyses. Special analyses will require more sample depending upon the tests requested. Where it is not possible to obtain a sufficiently large sample, special arrangements can be made with the laboratory personnel to perform the analyses in a given sequence so that the more important tests will be completed first.

6. SAMPLING METHODOLOGY AND FIELD RECORDS

The sampling personnel are usually in control of the sampling methodology and must be aware of how the particular details of the procedures and sampling apparatus (e.g. coring vs. dredge devices) may bias the results which are eventually obtained.

C - II. COLLECTION OF FISH SAMPLES FOR INORGANIC AND ORGANIC CONTAMINANTS ANALYSIS

1. ANALYTICAL TESTING CAPABILITIES

Fish samples, normally muscle tissue, are frequently analyzed for mercury or other metals by the Air Quality Section, pesticides by the Pesticide Section, and PCB's by the Organic Trace Contaminants Section. These analyses are very specialized and the following sample procedures should be closely followed.

2. SAMPLE PREPARATION AND SUBMISSION

- a) Details of species, length, weight, sex (if possible), location of catch, and organ (if other than muscle is being analyzed) must be neatly recorded on the submission sheet. As with all samples, the required analysis should also be indicated on the submission sheet.

b) FILLETING

Samplers are requested to submit fillets (rather than the whole fish) for analysis. Normally the analysis is carried out on tissue from the epaxial muscle (Figure 2) by making an incision with a stainless steel knife on the dorsal surface of the fish as shown (Incision #1). The epaxial muscle is then removed by cutting from the initial incision toward the tail (Filleting Incision #2) until a sufficient quantity of tissue is obtained. The muscle may be finally separated from the body by Incision #3. The skin should be removed from the sample.

It is important *not* to remove tissue from below the lateral line because of the high fat content in this region which makes PCB analysis impractical. *The sample should be frozen immediately after filleting and transported to the laboratory in this condition. This is the only acceptable preservation technique.* Samples collected for analysis of Hg or metals should

be submitted to the attention of Mr. Ray McVicars, while those for analysis of PCB's or pesticides should be brought to the attention of Mr. G. Rees.

c) SAMPLE SIZE

The minimum and preferred quantities of tissue required for each type of analysis are as follows:

	Absolute minimum (g)	Preferred (g)
Mercury	2	10
Other Metals	5	40
PCB, Pesticides	10	50

d) SAMPLE CONTAINERS

Individual samples collected for metals and mercury analysis may be placed in small plastic bags and then frozen. Clear identification with a sample code using a masking tape label is recommended, while the use of some variety of water-proof ink is a necessity.

Sample collected for PCB or pesticide analysis must be wrapped in solvent washed aluminum foil prior to freezing. Multiple washing of the foil and knife with hexane or acetone is a necessity. *Samples submitted in plastic bags for these analyses will not be accepted.* When both Hg and PCB's are required, submit the sample (frozen) in solvent washed foil.

3. OTHER CONSIDERATIONS

Analyses on tissues other than muscle is possible but can only be done by special arrangement. Any further queries should be directed to Dr. B. Loescher (248-3775), Air Quality Section, or Mr. G. Rees (248-3743), O.T.C.

D. LEGAL AND COMPLAINT SAMPLING

Sampling in connection with legal action naturally requires special care due to the influence this sampling may have on case outcome. Court cases are usually initiated to

determine legal responsibility for reported pollution events (stream, well contamination, vegetation or paint damage, etc.) and sampling must be conducted with this purpose in mind. In general, standard sampling methods as described previously may be used; however the following additional points and techniques should be fully read and understood before taking *any* legal samples.

D - I. WATER SAMPLES FOR CHEMICAL ANALYSIS

The following points should be precisely adhered to when collecting court case water samples requiring chemical analysis:

- a) The sampling area should be completely "walked", i.e. checked over at the time samples are taken. The sampler will then be completely familiar with the overall geographic "picture" (i.e. ALL possible contamination sources, unusual occurrences, and a "blank" sample location far enough away (upstream) that no contamination from the sources in question can influence it). Preparation of a sketch map of the area is recommended.
- b) The sampler should be careful to obtain samples at *all* possible contamination sources, not just the one in question. The observed contamination should be traced back to its source, and samples collected at key points to show continuity. In the case of an underground sewer, when the defendant or his official agent is unwilling to confirm continuity of flow of his wastes through the sewer, in front of a witness, the sampler should verify continuity by passing some small, identifiable floating object through the sewer, and recovering it at the outfall. Similarly a series of samples downstream is advised to show how the contamination effect persists. *A pre-requisite is a "blank" sample unaffected by the alleged pollution, (obtained upstream, or from a nearby well, etc.).*
- c) The sampler should obtain prior knowledge of exactly what type of contamination he is dealing with (i.e. what parameter(s) will be measured) and sample accordingly with respect to correct bottles, preservatives, etc.
- d) Legal samples must be analyzed in duplicate and thus it is recommended that at least *three times the normal volume* be submitted. Any remaining sample may then be used for further confirmation or presentation in court.

- e) *The actual sampling must be done in front of a witness who is willing to sign a witness affidavit and appear in court if necessary.*
- f) A complete record of exactly described sampling locations, time and date, bottle numbers, preservatives, etc., must be made. Submission sheets should accompany the samples in the normal manner. However, it is emphasized that the sample description and number on the bottle must *exactly* correspond to that on the sheet. If not, the certificate of analysis can be questioned, and may not be accepted as evidence.
- g) The sampler must be able to swear that the samples were in his possession and control before arrival at the laboratory, and not tampered with in any way. Locking the samples in the car trunk and delivering them directly to the official laboratory "analyst" is preferable. Otherwise, shipping boxes should be locked and the keys sent by a different route, normally registered mail, addressed to the "analyst" at the laboratory. The Toronto Laboratory Stores can provide special locks to return legal samples, for which the main Laboratory analysts (only) have keys.
- h) Enquiries with regard to sampling for chemical analysis, should be directed to C. Simpson, Project Scientist, Resources Road, Toronto, (248-3064), and for microbiological analysis to J. A. Clark, Supervisor, Resources Road, (248-3008).

D - II. SAMPLING FOR PARTICLE IDENTIFICATION

In many instances, generally arising from citizens' complaints, it becomes necessary for field personnel to collect samples for constituent identification by means of microscopic, X-ray diffraction, electron probe and other techniques. The types of material normally encountered are visible solids present in air or water, that are a cause of nuisance or concern to the complainant. To facilitate the collection of these special samples, a sampling kit is available from the Laboratory Services Branch for each district office in the province. Supervisory personnel should obtain this and maintain this kit, which also includes more detailed sampling instructions. General guidelines to be used for sampling are given below.

1. AIR SAMPLES

Dust fallout is the most frequent cause of complaints. In these cases, the best method of obtaining a sample for particle identification is by brushing the dust into the specimen container (*47 mm plastic petri dish*) using a clean brush. The petri dish should not be sealed with any type of tape. The closed dish does not easily come apart. Dust, adhering strongly to any surface, may be removed by lifting it by means of scotch tape and also with the conductive copper tape provided in the kit. Whenever tape has been used for sampling, it should be protected by means of the covering strip which comes with the tape or attached to a glass microscope slide. Under *no conditions* should the tape be folded on itself. When sampling suspected soot fallout (especially oil soot), the use of tape is not advisable, as the pressure used in collecting it, often destroys the identifying characteristics. In such cases it is better to remove a small paint section from outside window sills, shutters, etc. Plant leaves, eavestroughing, bird baths, furnace and air conditioner filters often act as collectors of particulate fallout. Where the fallout occurs consistently, aluminum weighing dishes, wetted with a glycerine-water mixture, can be used as miniature dustfall jars. These can be attached to a suitable vertical surface by means of a thumb tack.

As a general rule, samples should not be collected from nonstationary objects such as automobiles, since the source of the dust may then be in question. Damage to automobile paint or house sidings is generally caused by very acidic or basic materials attacking the paint surface. Such types of fallout should be tested on the spot using pH indicator paper. It is often difficult to remove a representative sample from such surfaces, and on-site inspection by laboratory staff may be necessary to determine the cause of the damage.

Heavy dustfall onto snow should be sampled by scooping the snow into a large-mouth glass or plastic bottle in such a way as to maximize the amount of particulate material obtained and prevent any possible contamination from underlying soil.

All samples collected as a result of air pollution complaints should be accompanied by the analytical request and inspection report forms, and forwarded to Mr. D. Sturgis, Air Quality Section, 880 Bay Street, Toronto. These forms should provide all the information required to make a proper assessment of the situation.

A sketch map of the area is strongly recommended. If insufficient space is available on the form(s), add further sheets as necessary. Results will be reported on the Sample Analysis Report Form.

2. WATER SAMPLES

Water samples that require identification of the suspended solids, may be sent to the laboratory in any of the standard containers. A few milligrams of material are usually sufficient for microscopic analysis, although for complex mixtures requiring multi-instrumental analyses, up to a half gram of material would be preferable. In cases where preconcentration of the suspended material is required in the field, this can be done by means of the filter-syringe assembly supplied in the kit. Several syringe aliquots can be filtered before the filter gets clogged. Samples collected from water systems, water filtration plants, solid waste disposal, land fill sites, etc., should be sent along with the usual sample submission forms to Dr. J. Hipfner, Air Quality Section, Resources Road, Rexdale.

3. FURTHER INFORMATION

For further information regarding collection of samples for identification purposes or interpretation of results, please call Dr. J. Pimenta, Physical Methods Section, Resources Road, (248-7101). In certain circumstances field assistance by laboratory staff may be provided.

D - III. SAMPLING FOR GAS DAMAGE COMPLAINTS

In cases of suspected sulphide gas damage, the stained surface such as paint work, should be accompanied with an unstained sample, if available. Information as to the manufacturer and type of paint should also be obtained. Tarnishing of silverware, electrical contacts are usual indications of the presence of sulphide gases in the air. Where any type of damage due to corrosion has occurred (aluminum sidings, automobiles, wire fences), it is best to have laboratory staff inspect the damage and collect the sample for analysis. Special portable static samplers or gas detectors are available from the laboratory for sampling a number of gases. Contact G. S. Rees, Physical Methods Laboratory, (248-7101) for information.

TABLE I - ANALYTICAL TESTING CAPABILITIES

LABORATORY SERVICES BRANCH

CODE - W - Water Quality Section

A - Air Quality Section

O - Organic Trace Contaminants Section

P - Physical Methods Section

L - London Regional Laboratory

T - Thunder Bay Regional Laboratory

K - Kingston Regional Laboratory

A. MAJOR IONS								C. METALS							
Parameter	W	A	O	P	L	T	K	Parameter	W	A	O	P	L	T	K
Alkalinity	X				X	X	X	Aluminum		X					
Calcium	X	X			X	X	X	Arsenic		X				X	
Chloride	X	X		X	X	X	X	Barium		X					
Conductivity	X				X	X	X	Boron		X					
Hardness	X				X	X	X	Cadmium		X				X	
Magnesium	X	X			X	X	X	Chromium		X					
Potassium	X	X			X	X	X	Cobalt		X				X	
Silicates - Reactive	X				X			Copper		X				X	
Sodium	X	X			X	X	X	Iron (Total)	X	X			X	X	X
Sulphate	X	X			X	X		Lead		X				X	
								Lithium		X					
								Manganese	X					X	
								Mercury		X				X	
								Molybdenum		X					
								Nickel		X				X	
								Selenium		X					
								Silver		X					
								Strontium		X					
								Titanium		X					
								Uranium		X					
								Vanadium		X					
								Zinc		X				X	
B. NUTRIENTS															
Parameter	W	A	O	P	L	T	K								
Ammonia	X	X			X	X	X								
Nitrate	X	X			X	X	X								
Nitrite	X				X	X	X								
Orthophosphate (filtered reactive)	X				X	X	X								
Total Kjeldahl	X				X	X	X								
Total Phosphorus	X	X		X	X	X	X								

TABLE I - ANALYTICAL TESTING CAPABILITIES - LABORATORY SERVICES BRANCH

CODE - W - Water Quality Section

A - Air Quality Section

O - Organic Trace Contaminants Section

P - Physical Methods Section

L - London Regional Laboratory

T - Thunder Bay Regional Laboratory

K - Kingston Regional Laboratory

D. ORGANIC PARAMETERS							E. OTHER PARAMETERS							
Parameter	W	A	O	P	L	T	Parameter	W	A	O	P	L	T	K
Anionic Detergents	X				X	X	Acidity	X				X	X	X
Benzene			X				Asbestos (in Air or Water)				X			
Benzene Soluble Organics			X				Chlorophyll	X					X	
Benzo (a) Pyrene			X				Cyanide		X					
Benzo (k) Fluoranthone			X				Fluoride	X	X			X		
Biochemical Oxygen Demand	X				X	X	Loss on Ignition	X	X		X			
Carbon - Free (elemental)				X			Nitritotriacetic Acid			X				
Carbon Dioxide	X						Particle Size Analysis		X					
Carbon - Inorganic	X						Particle Size by Microscopy				X			
Carbon - Total	X			X			Particulate Identification				X			
Carbon - Total Organic	X						(Complaint Samples)							
Chemical Oxygen Demand	X	X			X	X	pH	X	X			X	X	X
Colour - Apparent							Plasticity (Sediments)		X					
Foams			X				Settleability	X						
Methane			X				Sieve Analysis				X			
Oils			X				Silicon		X		X			
Other Aliphatic Hydrocarbons			X				Sludge Volume Index	X						
Petroleum - Hydrocarbons			X				Solids - Filtered	X				X	X	X
Phenolics - Reactive	X			X	X	X	Solids - Ignited	X				X	X	X
Resins & Fatty Acids				X			Solids - Suspended	X				X	X	X
Solvent Extractables			X				Sulphide		X			X		
Tannins & Lignins			X				Sulphur - Total		X		X			
Toluene			X				Turbidity	X				X	X	X
Tracer Dyes			X				Vinyl Chloride			X				
Vinyl Chloride			X											
Volatile Acids	X				X	X								
Volatile Organohalides			X											
Xanthates			X											
Xylenes			X											
Pesticides*														

*All Pesticide and Herbicide analyses are performed by the Pesticides Section, Resources Road, Toronto.

TABLE II - SPECIFIC PARAMETER INFORMATION

	Parameter	Container	Preservation Technique	Minimum Volume Required for each Parameter (ml)	Comment (Syn = Synonym)
U N I T A M A J O R I O N S	Alkalinity	Glass or Plastic	None	50	Syn = Specific Conductance
	Calcium	" "		40	
	Chloride	" "		50	
	Conductivity	" "		75	
	Hardness	" "		50	
	Magnesium	" "		40	Syn = Silica
	Potassium	Plastic or Glass		40	
	Silicates - Reactive	Plastic only		50	
	Sodium	Glass or Plastic		40	
	Sulphate	" "		50	
U N I T B N U T R I E N T S	Ammonia Nitrogen (Filtered)	Glass or Plastic (polystyrene not linear polyethylene)	Freeze or Refrigerate	75	Syn = Nitrogen - Ammonia
	Nitrate Nitrogen (Filtered)				Syn = Nitrogen - Nitrate
	Nitrite Nitrogen (Filtered)				Syn = Nitrogen - Nitrite
	Orthophosphate				Syn = Phosphorus - Filtered Reactive
	Nutrient - Total Kjeldahl				
	Phosphorus - Total				Syn = Total Phosphorus

TABLE II - SPECIFIC PARAMETER INFORMATION (Cont'd)

	Parameter	Container	Preservation Technique	Minimum Volume Required for each Parameter (ml)
U N I T C M E T A L S	Aluminum	Plastic or Glass ²	HNO ₃ to pH of 1 (approximately 20 drops per bottle) ¹	100-unless preconcentration required
	Barium			
	Cadmium			
	Chromium			
	Cobalt			
	Copper			
	Iron			
	Lead			
	Lithium			
	Manganese			
	Molybdenum			
	Nickel			
	Selenium			
	Silver			
	Strontium			
	Titanium			
	Uranium			
	Vanadium			
	Zinc			
	Arsenic	Plastic or Glass ²	None	50
	Boron	Plastic only	None	100
	Mercury	Glass only	HNO ₃ to pH of 1 + KMnO ₄ to maintain slight purple colour/bottle	176 ³

¹ Nitric acid preservative should be added AFTER the sample is placed in the bottle.

² Acid washed plastic containers are recommended for ultra-trace analysis; foil cap liners of glass bottles used for routine samples may cause contamination, and plastic lined caps are recommended.

³ A special 176 ml sample bottle similar to the microbial analysis type is usually provided for Hg samples.

TABLE II - SPECIFIC PARAMETER INFORMATION (Cont'd)

	Parameter	Container	Preservation Technique	Minimum Volume Required for each Parameter (ml)	Comment (Syn = Synonym)
U N I T D O R G A N I C P A R A M E T E R S	Anionic Detergents	Glass	Refrigerate	100	Syn = L.A.S., Linear Alkyl Sulfonates, Methylene Blue Active Substances, Detergents.
	Biochemical Oxygen Demand	Glass		500	Syn = BOD ₅
	Carbon - Total Organic	Glass or Plastic		50	Syn = TOC
	Carbon - Inorganic	Glass or Plastic		50	Syn = IC
	Carbon Dioxide	Special *		*	Syn = Free CO ₂ . Special sampling required.
	Chemical Oxygen Demand	Glass		25	Syn = COD
	Colour - Apparent	Glass		75	Syn = Apparent Colour
	Pesticides	Glass only		900	Syn = Chlorinated Hydrocarbons
	Petroleum Hydrocarbons	Glass only		900	Syn = Hydrocarbons
	Solvent Extractables	Glass		900	Syn = Ether solubles
	Tannins and Lignins	Glass		200	
	Volatile Acids	Glass		25	
* CO ₂ samples are to be carefully transferred from the sampling device into the bottom of a leak-proof glass stoppered container so as to prevent splashing (syphon); after copious overflow the bottle must be stoppered so that no air bubbles are present in the container and rushed to a laboratory.					

TABLE II - SPECIFIC PARAMETER INFORMATION (Cont'd)

	Parameter	Container	Preservation Technique	Minimum Volume Required for each Parameter (ml)	Comment (Syn = Synonym)
UNIT OTHERS	Acidity	Glass or Plastic	None	50	Contact Water Quality Section if there are any questions.
	Chlorophyll	Field Filtration Required	1 ml 10% $MgCO_3$ per litre of sample prior to filtration	500	
	Cyanide	Glass or Plastic	1 ml 50% NaOH per bottle	500	
	Fluoride	Glass or Plastic	None	50	
	Nitritotriacetic Acid	Glass only	2 ml Formaldehyde per 20 oz bottle	600	
	Particle Size Analysis (Sediment)	Glass or Plastic	None	100 g.	Syn = NTA
	pH	Glass or Plastic	None	25	
	Phenolics - Reactive	Special	Provided	150 ¹	
	Plasticity (Sediment)	Glass or Plastic	None	100 g	
	Resins & Fatty Acids	Glass only	Refrigerate + 2 drops 1 N NaOH per bottle	900	
PARAMETERS	Settleability	Glass	None	900	One bottle should be submitted for this test exclusively, labelled Resin Acids. Syn = Fatty Acids
	Sludge Volume Index	None	None	None	Calculated Parameter

A special 176 ml sample bottle similar to the microbial type (with preservative added) is usually provided for samples requiring analysis for phenolics. A culture tube sample container with preservative is also available.

TABLE II - SPECIFIC PARAMETER INFORMATION (Cont'd)

	Parameter	Container	Preservation Technique	Minimum Volume Required for each Parameter (ml)	Comment (Syn = Synonym)
U N I T E O T H E R P A R A M E T E R S	Solids - Filtered	Glass	Refrigerate	75	} Consult laboratory staff if low level analysis required.
	Solids - Ignited	Glass	Refrigerate	200	
	Solids - Suspended	Glass	Refrigerate	200	
	Sulphide	Glass or Plastic	Zinc Acetate* + Sodium Carbonate	900	Consult Lab. personnel prior to sampling for sulphide.
	Turbidity	Glass or Plastic	Keep in darkness	50	
	Vinyl Chloride	Special	Keep in darkness	10	Special "hypovial" sample container available from the Toronto laboratory.

* 2 ml of 2N Zinc Acetate followed by dropwise addition of 5% Sodium Carbonate solution until precipitation complete.

TABLE III

COMMON PARAMETER GROUPS

Group	Routine Parameters	Possible Additional Parameters	Bottle Type + Volume Required	Preservation Technique	Comment
Routine Drinking	Hardness, Alkalinity, Chloride, Iron, pH, Conductivity	Colour Turbidity Fluoride	1 x 32 oz, 1 x 20 oz, or 1 x 1 litre Glass or Plastic Bottle	Refrigerate	Phenol is included in this group but requires a special bottle and preservative. Chloramine, H ₂ S may be included but should be measured in the field.
Taste and Odour Group	Hardness, Alkalinity, Chloride, Nitrate, Iron, pH, Conductivity, Manganese, Kjeldahl, Nitrogen, TOC		1 x 32 oz, 1 x 20 oz, or 1 x 1 litre Glass Bottle	Refrigerate	
Ionic Balance Group	Calcium, Magnesium, Sodium, Potassium, Hardness, Alkalinity, Sulphate, Chloride, Nitrate	pH, Iron, Conductivity	1 x 20 oz, 1 x 32 oz, or 1 litre Glass or Plastic Bottle	Refrigerate	

TABLE III

COMMON PARAMETER GROUPS (Cont'd)

Group	Routine Parameters	Possible Additional Parameters	Bottle Type + Volume Required	Preservation Technique	Comment
Well Characterization Group (for Domestic or Industrial Use)	Sodium, Potassium, Hardness, Alkalinity, Sulphate, Chloride, Nitrate, Conductivity, Manganese, Kjeldahl Nitrogen, Total Organic Carbon	Total Phosphorus, Iron, Calcium, Magnesium Fluoride	2 x 32 oz, 2 x 20 oz, or 2 x 1 litre Glass or Plastic	Refrigerate	H ₂ S may be included but should be measured in the field.
Sewage Group	BOD, Ammonia, Nitrate, Phosphorus, Kjeldahl Nitrogen, Total Phosphorus, Suspended Solids, Dissolved Solids	COD, Nitrite	2 x 20 oz, 1 x 32 oz, or 1 x 1 litre Glass Bottle	Refrigerate	
Trace Metal Group	Cadmium, Copper, Iron, Lead, Zinc		1 x 32 oz Plastic Bottle	20 drops HNO ₃	

TABLE IV

SAMPLE BOTTLE REQUISITION

Order Number	Number and Size of Bottles	Description
Pack # 1	6 x 1 litre	glass, packed in styrofoam
Pack # 1A	6 x 1 litre	glass, packed in styrofoam (for P.C.B.)
Pack # 1S	6 x 1 litre	glass, packed in styrofoam (with Special Cap-non metal)
Pack # 2	1 x 1 litre	glass, packed in styrofoam
Pack # 2A	2 x 1 litre	glass, packed in styrofoam (with Special Cap-non metal)
*Pack # 3	8 x 1 litre	glass, packed in styrofoam
*Pack # 3P	8 x 1 litre	glass, packed in styrofoam (for P.C.B.)
*Pack # 3S	8 x 1 litre	glass, packed in styrofoam (with Special Cap-non metal)
Pack # 4	8 x 32 oz.	glass, Wide Mouth (approximately 1 litre)
Pack # 5	16 x 16 oz.	glass, Wide Mouth (approximately 1/2 litre)
Pack # 6B	2 x 6 oz.	glass, Bacti blue (Sterile) (approximately 1/5th litre)
Pack # 6P	2 x 6 oz.	glass, Phenol Green (approximately 1/5th litre)
Pack # 6T	2 x 6 oz.	glass, Thio Red (approximately 1/5th litre)
Pack # 7B	4 x 6 oz.	glass, Bacti Blue (Sterile) (approximately 1/5th litre)
Pack # 7P	4 x 6 oz.	glass, Phenol Green (approximately 1/5th litre)
Pack # 7T	4 x 6 oz.	glass, Thio Red (Sterile) (approximately 1/5th litre)
Pack # 8B	72 x 6 oz.	glass, Bacti Blue (Sterile) (approximately 1/5th litre)
Pack # 8T	72 x 6 oz.	glass, Thio Red (Sterile) (approximately 1/5th litre)
Pack # 9	8 x 32 oz.	plastic, Wide Mouth, 63 mm cap size (approximately 1 litre)
Pack # 10	8 x 32 oz.	plastic, Wide Mouth, Acid Wash (approximately 1 litre)
Pack # 11	8 x 16 oz.	plastic, Wide Mouth (approximately 1/2 litre)
Pack # 12	24 x 6 oz.	plastic, Polystyrene (approximately 1/5th litre)
Pack # 13	12 x 16 oz.	plastic, Polystyrene (approximately 1/2 litre)

* PACK #3, 3P, 3S WILL BE DISCONTINUED ON/OR ABOUT SEPTEMBER 1977.

NOTE: THE 32 OZ. AND 20 OZ. GLASS BOTTLES WITH NARROW MOUTH ARE NO LONGER AVAILABLE.

TABLE V

<u>Additional Analytical Testing Capabilities</u>	-	<u>Laboratory Services Branch</u>
Parameter Specific to Air Quality Assessment ⁺	-	all tests performed by the Air Quality Section, except as noted.
Freons ⁺		
Dust fall		
Sulphation Rate		
Fluoridation Rate		
Total Suspended Particulates		

+ Performed by the Organic Trace Contaminants Section

Other Air Quality Assessment parameters which may be used to test other media (i.e. water, vegetation, etc.) are found in Table I.

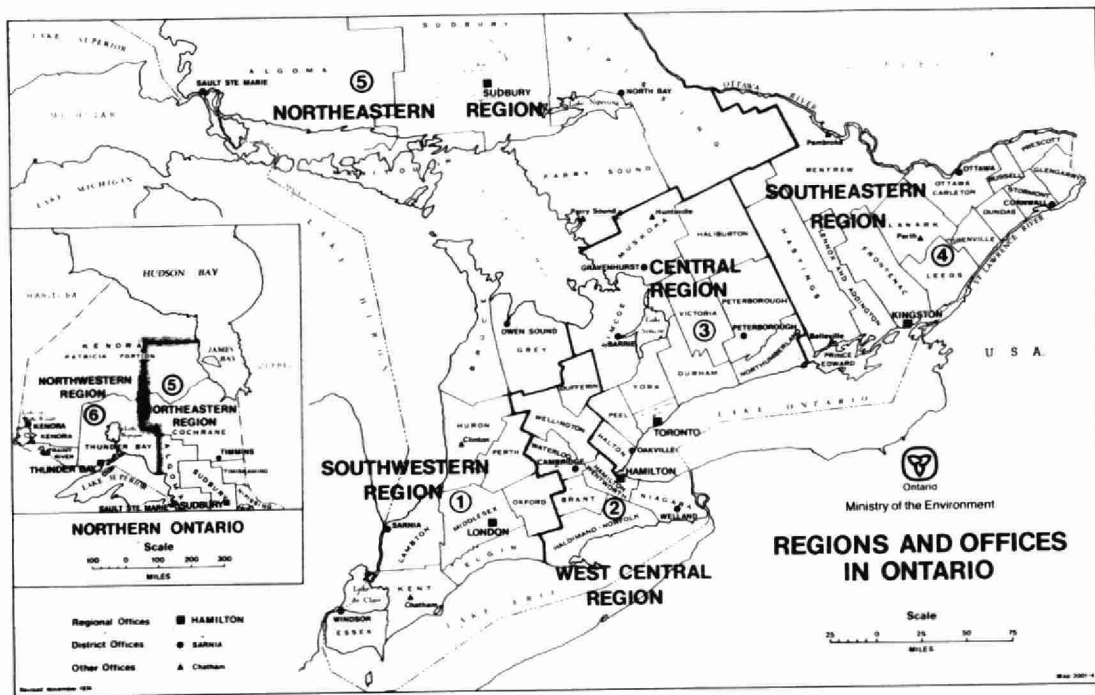
TABLE VI

ADDITIONAL TESTS PERFORMED BY THE SEDIMENT AND SOILS LABORATORY*

TESTS DESIGNATION	SAMPLE SIZE	EXTRACTING SOLUTION	PARAMETERS
Water Soluble	5 - 10 g dry weight	Water (5 : 1 = distilled water : solid)	Chloride Sulphate Ammonia Nitrite Nitrate Phosphate Fluoride BOD Conductivity
Acid Soluble	1 - 5 g	a) 0.5N H_2SO_4 b) H_2SO_4 -Persulphate digestion c) HCl (concentrated)	{ Phosphorus (inorganic) { Total Phosphorus { Total Nitrogen { Sodium after ignition { Potassium at 550 C.
Base Soluble	1 - 5 g dry weight	Non-apatite P - 0.1N NaOH Bicarbonate available P - 0.5N $NaHCO_3$	Phosphorus (inorganic)
Leaching Analysis	This type of analysis could involve any of the parameters above or those listed in Table I.		

* Please note that many of the above parameters require laboratory consultation before the sample is submitted for analysis. Analyses of a special nature may be arranged to suit the requirements of particular programmes.

FIGURE 1



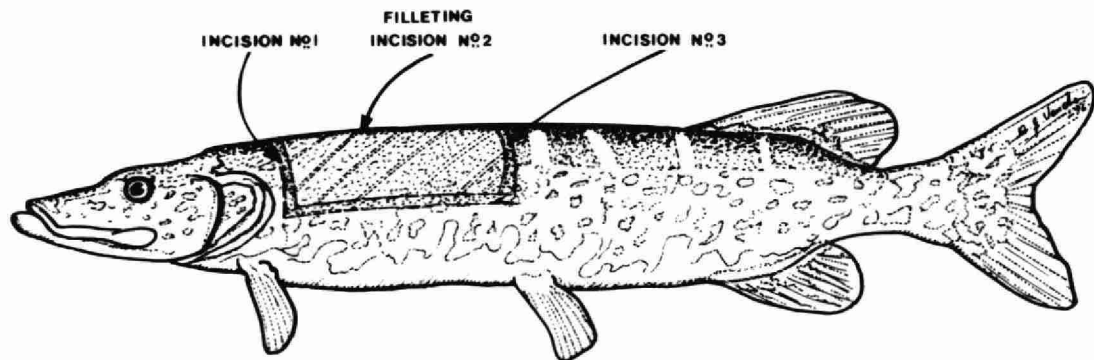


Figure 2



Environment Ontario
Laboratory Library
125 Resources Rd.
Etobicoke, Ontario M9P 3V6
Canada

APPENDIX I

Enquiries regarding sampling and test procedures and the status of outstanding samples should be directed to the appropriate individuals listed below.

WATER QUALITY SECTION

Sewage and Sludge Sampling and Analysis	Joan Crowther	416-248-3022
Well and Drinking Water Sampling and Analysis	Mike Rawlings	248-3024
River and Lake Sampling and Analysis	Fred Dieken	248-3064
Precipitation, Snow and Low Level Nutrients	Dean Jeffries	248-3003
Legal Samples	Chuck Simpson	248-3064
Status of Samples through Lab	Gwen Nicolls	248-3064

AIR QUALITY SECTION

Metals, Fish, Legal Samples (Water)	Barry Loescher	416-248-3775
Sediments	Frank Darcel	248-3775
Spectrographic Scans	M. Moselhy	248-3029
HiVol, Dustfall, Legal Samples	Brian Foster	965-1574
Vegetation and Soils (Air Fallout)	Ron Wills	965-1574
Status of Samples through Lab	Cliff Lee	248-3775

ORGANIC TRACE CONTAMINANTS SECTION

Oil and Petroleum Hydrocarbons, Organic Characterization Court Cases	George Wyhovszky	248-3469
Halofoms in Water, PAH Organic, Air Sampling	Ed Adamek	248-3755
Status of Samples through Lab	George Wyhovszky	248-3469

PHYSICAL METHODS SECTION

Electron Microscopy (Asbestos)	Tom Pang	416-248-7101
Complaint Type Samples requiring Elemental Identification, X-ray fluorescence	Jim Pimenta	248-7101
Sampling Particulate Identification	Aston Hinds	248-7101

APPENDIX I (Cont'd)

PESTICIDES SECTION

PCB and Chlorinated Hydrocarbons
Herbicides and Triazines
Organophosphates

George Crawford
Joe Osborne
Pat Baulu

416-248-3743
248-3743
248-3743

MICROBIOLOGY SECTION

Drinking Water
Sewage and Industrial Wastes
Great Lakes
Rivers
Recreational Lakes
Status of Outstanding Samples and Tests

Jim Clark
David Rokosh
Mike Young
Anser Qureshi
George Hendry
Charlotte Scheibelt

416-248-3008
248-3008
248-3008
248-3008
248-3008
248-3008

LONDON REGIONAL LABORATORY

Chief Laboratory Services
Chemistry
Microbiology

Dave Glutek
Walter Cook
Gary Palmateer

519-681-3600
681-3600
681-3600

THUNDER BAY REGIONAL LABORATORY

Chief Laboratory Services
Chemistry
Microbiology

Al Perras
Mark Mazurski
Stuart Irwin

807-475-1275
475-1275
475-1275

KINGSTON REGIONAL LABORATORY

Chief Laboratory Services
Scientist

Stan MacBeth
Art Ley

613-549-4000
549-4000

QUALITY CONTROL

Don King

416-248-3064

SHIPPING AND RECEIVING, Central Stores,
Sample Bottle Supply

Hilda Liddell

416-248-7134
or-248-3051